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Process for purifying caprolactam

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PROCESS FOR PURIFYING CAPROLACTAM

The invention relates to a process for purifying caprolactam, said process comprising
(a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst, and
(b) distilling at least a portion of said hydrogenated caprolactam in a distillation column.

Impure caprolactam, prepared by for example Beckmann rearrangement of cyclohexanone oxime, is subjected to a number of purification steps to obtain caprolactam of the purity required for polymerisation to nylon 6. A possible purification step is the hydrogenation that can be carried out to hydrogenate unsaturated organic compounds that can be present in the impure caprolactam. The presence of these unsaturated compounds is disadvantageous because they can impair the physical-mechanical properties of the nylon-6 made by polymerizing caprolactam. The saturated organic compounds formed by hydrogenation do not adversely influence these physical-mechanical properties of the nylon-6 and moreover these compounds are more easily removed in for example a distillation following the hydrogenation.

Such a process is described in EP-A-138241. In the process as described in EP-A-138241 caprolactam is mixed with water, the so obtained aqueous caprolactam mixture is subsequently hydrogenated in the presence of a Raney nickel catalyst (example I) or a nickel on SiO_2 hydrogenation catalyst (example II) suspended in the aqueous caprolactam mixture to be purified. The hydrogenation catalyst is subsequently filtered off and water is removed by distillation at atmospheric pressure. The remaining product is distilled at a pressure of 0,8 kPa and a temperature of 123°C.

It has surprisingly been found that the caprolactam obtained in such process still has a high PAN number.

The object of the invention is therefore a process for the purification of caprolactam wherein the PAN number of the obtained caprolactam is further reduced.

The object of the invention is achieved in that the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 3$, wherein $\Delta PAN_{Ni} = \Delta PAN - \Delta PAN_{Ni=0}$,

ΔPAN = increase of the PAN number of caprolactam during distilling.
$\Delta PAN_{Ni=0}$ = increase of the PAN number of caprolactam during distilling under the same conditions but in a distillation column free of nickel.

Preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN_{Ni} \leq 2$. More preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN_{Ni} \leq 1$.

In a preferred embodiment, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN \leq 3$. More preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN \leq 2$. Even more preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN \leq 1$.

As used herein, the increase of the PAN number of caprolactam during distilling refers to the PAN number of caprolactam leaving the distilling minus the PAN number of caprolactam entering the distilling. As used herein, the PAN number (permanganate absorption number) is determined in accordance with ISO standard 8660. The PAN number of caprolactam is a measure of the oxidizable impurities content of caprolactam. A higher PAN number means that a higher amount of oxidizable impurities is present.

The process of the present invention also relates to a process for purifying caprolactam, said process comprising
(a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst,
(b) distilling at least a portion of the hydrogenated caprolactam in a distillation column, characterized in that the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN \leq 3$, wherein ΔPAN = increase of the PAN number of caprolactam during distilling. Preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN \leq 2$. More preferably, the amount of nickel in said distillation column is sufficiently low such that $\Delta PAN \leq 1$.

It has surprisingly been found that the quality of the caprolactam deteriorates, in particular the PAN number increases, during said distilling in particular when having distilled high amounts of hydrogenated caprolactam. The process of the invention provides a process in which it is possible to distill a higher amount of hydrogenated caprolactam while the deterioration of the quality of caprolactam in said distilling remains the same or is even reduced.

It has surprisingly been found that reducing the amount of nickel in the distillation column results in less deterioration of the quality of caprolactam during

said distilling, in particular reducing the amount of nickel in the distillation column results in that the PAN number of caprolactam during said distilling increases to a lesser extent. Reducing the amount of nickel in the distillation column to such an amount that the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 3$ is especially advantageous in case the distilling of the hydrogenated caprolactam is performed continuously. In such continuous distillation hydrogenated caprolactam is supplied continuously to the distillation column and the distilled products are continuously withdrawn from the distillation column. In order to reduce the necessity of reducing the amount of nickel by for example cleaning the distillation column and thus in order to reduce the necessity of interruptions of a continuous distillation, it is important that high amounts can be distilled without high deterioration of the quality of the product to be distilled. The process of the present invention therefore especially relates to a process in which said distilling is performed continuously.

It has surprisingly been found that the presence of nickel in the distillation column has proved to be the cause of the deterioration of the quality of the caprolactam during said distilling inasmuch as nickel readily convert caprolactam into so-called unsaturated lactams (UCL) mainly into the compound denoted on the formula sheet as UCL-1. On the formula sheet, four of such unsaturated lactams with their structural formulae are drawn and denoted as UCL-1, UCL-2, UCL-3 and UCL-4. The PAN number is a measure of the oxidizable impurities content in caprolactam. The UCL's inter alia belong to the oxidizable impurities.

It was not to be expected that the quality deterioration during distilling of the hydrogenated caprolactam was caused to a large extent by the presence of nickel in the hydrogenated caprolactam fed to said distilling. It has in fact been found that, although customary techniques are applied for separating catalyst particles from the hydrogenated caprolactam, the hydrogenated caprolactam, obtained after such separation still comprises nickel. In addition, formation of unsaturated lactams may be caused by other types of chemical reactions, like for example oxidation, and/or may be caused by the presence of impurities in compounds used in the various chemical steps to produce caprolactam. Moreover, nickel is not generally known to form unsaturated lactams from caprolactam under the usually applied distillation conditions.

The hydrogenated caprolactam entering said distilling usually has a PAN number of between 2 and 5. The higher the PAN number of the hydrogenated caprolactam entering said distilling, the lower the tolerated increase of the PAN number of caprolactam during said distilling to obtain caprolactam of the preferred quality.

Depending on the PAN number of the hydrogenated caprolactam entering said distilling, the tolerated ΔPAN_{Ni}, preferably the tolerated ΔPAN is at most 3, preferably at most 2 and more preferably at most 1.

In the process of the invention, caprolactam containing unsaturated organic compounds is subjected to a hydrogenation in the presence of a heterogeneous nickel containing catalyst resulting in that unsaturated compounds are hydrogenated. Examples of heterogeneous nickel containing catalysts are Raney nickel catalysts or supported nickel catalysts. Suitable supported nickel catalysts generally have a nickel content from 5 to 80% by weight, based on metal content and carrier. Besides nickel the catalyst may also contain activating additives such as zirconium, manganese, copper, molybdenum, iron or chromium, for example in amounts of from 1 to 20% by weight, based on the amount of nickel employed. The carriers used advantageously are alumina, silica, diatomaceous earth or activated carbon. Particularly advantageous carriers are alumina and silica.

The hydrogenation may be carried out as a slurry phase process or in a fixed bed reactor with the catalyst being fixed in the reactor or with a fluidized bed catalyst. Preferably the hydrogenation is carried out as a slurry phase process or in a fixed bed reactor with the catalyst being fixed in the reactor. In case the hydrogenation is carried out as a slurry phase process, the hydrogenation is preferably carried out in a stirred tank reactor in which the catalyst particles are suspended in the caprolactam to be hydrogenated in a pulverulent or granular form. In such slurry phase hydrogenation, the catalyst particles and the hydrogenated caprolactam are separated in an additional process step after the hydrogenation reaction step. Usually, such separation is effected by means of filtration. It has surprisingly been found that, although customary separation techniques like for example filtration are applied for separating catalyst particles from the hydrogenated caprolactam, the hydrogenated caprolactam, obtained after such separation, still contains nickel. An example of a customary separation technique for separating catalyst particels from hydrogenated caprolactam is cake filtration using for example textiles woven of cotton or synthetic fibers. An example of a suitable filter for separating catalyst particles from hydrogenated caprolactam is a Funda filter.

More preferably, the hydrogenation is effected in a fixed-bed reactor with the catalyst being fixed in the reactor. It has surprisingly been found that effecting the hydrogenation in a fixed-bed reactor with the catalyst being fixed in the reactor also results in the presence of nickel in the hydrogenated caprolactam. The hydrogenation

is however preferably effected in a fixed-bed reactor with the catalyst being fixed in the reactor because the additional step of separating the catalyst and the hydrogenated caprolactam can be dispensed with. An example of a possible fixed-bed reactor is the trickle-phase reactor.

The hydrogenation temperature is generally between 20 and 160 °C. As a rule a not too low temperature will be chosen, for at a low temperature the reaction time is longer. The temperature is as a rule not too high because high temperatures have a negative influence on the caprolactam quality. The temperature therefore preferably is between 60 and 130 °C. The hydrogenation pressure may be between 0.1 and 3 MPa. Preferably the pressure is between 0.2 and 2 MPa. The hydrogenation can be carried out as for example described in EP-A-411455 or EP-A-635487.

Preferably, the caprolactam which is subjected to the hydrogenation is dissolved in a solvent, preferably in water. The caprolactam which is subjected to the hydrogenation preferably contains between 10 and 98 wt.% caprolactam and preferably between 2 and 90 wt.% water.

In case the hydrogenated caprolactam contains a substantial amount of water, for example more than 2 wt.% water relative to the total amount of hydrogenated caprolactam, water is preferably separated from the hydrogenated caprolactam prior to said distilling of the hydrogenated caprolactam. Separating water from the hydrogenated caprolactam may be effected in any suitable way, for example evaporating or distilling at reduced pressure.

The hydrogenated caprolactam entering said distilling contains caprolactam, water, light components (among others unsaturated lactams) and heavy components. As used herein, light respectively heavy components are components having a boiling point lower respectively higher than the boiling point of caprolactam. Said distilling aims to recover caprolactam from the hydrogenated caprolactam and to further purify the hydrogenated caprolactam. Said distilling is preferably performed in a vacuum distillation column. Preferably, the applied pressure at the top of the distillation column is between 0.2 and 5 kPa and the bottom temperature is preferably between 110 and 180 °C. It has been found that the process of the invention is especially advantageous when said distilling of the hydrogenated caprolactam is effected in a distillation column in which the caprolactam of the hydrogenated caprolactam has a residence time of at least 5 minutes, for instance between 5 and 50 minutes. With the residence time is meant the time period between feeding the hydrogenated

caprolactam comprising a certain amount of caprolactam and withdrawing that amount of caprolactam from the distillation column. More in particular, the process of the present invention is especially advantageous when distilling the hydrogenated caprolactam is effected in a distillation column having a bottom temperature of between 110 and 180 °C and in which the caprolactam of the hydrogenated caprolactam has a residence time of at least 5 minutes. An example of a distillation column in which the residence time of caprolactam is at least 5 minutes is a distillation column with sieve plates or a packing to which the hydrogenated caprolactam is fed above or on the same level as the sieve plates or packing.

The process according to the invention can be carried out in a variety of embodiments. All embodiments will aim to be able to increase the amount of hydrogenated caprolactam which can be distilled while the amount of nickel in the distillation column remains sufficiently low such that $\Delta PAN_{Ni} \leq 3$, preferably $\Delta PAN \leq 3$. To achieve this, it is important that the concentration of nickel in the hydrogenated caprolactam fed to said distilling should be as low as possible. In some cases, if the cost of (substantial) removal of the nickel from the hydrogenated caprolactam to be distilled is higher than the economic losses resulting from build-up of nickel in the distillation column, it may be acceptable for a small amount of nickel to be present in the hydrogenated caprolactam fed to the distillation column. The term parts per million is understood to mean the ratio consisting of grams of nickel per one million grams of caprolactam in a mixture comprising nickel and caprolactam.

In a first embodiment, the amount of nickel in the distillation column is kept sufficiently low such that $\Delta PAN_{Ni} \leq 3$, preferably $\Delta PAN \leq 3$. by cleaning the distillation column with sufficient frequency. Cleaning the distillation column is preferably effected by interrupting the distilling of the hydrogenated caprolactam, feeding an acidic solution in which nickel has a high solubility, like for example nitric acid, and rinsing the column with said acidic solution. Preferably, the acidic solution is an aqueous acidic solution.

In a second and preferred embodiment, the hydrogenated caprolactam is substantially freed (in a separate operation) of nickel prior to said distilling. In this embodiment, the process of the present invention further comprises, prior to said distilling and after said hydrogenating and in case of a slurry phase hydrogenation after having separated catalyst particles from the hydrogenated caprolactam, separating nickel from the hydrogenated caprolactam. In case water is separated from the hydrogenated caprolactam prior to said distilling of the

hydrogenated caprolactam, the operation to separate nickel from the hydrogenated caprolactam is preferably effected after having separated water and prior to said distilling. Preferably separating from the hydrogenated caprolactam is effected such that after said separating the amount of nickel in the hydrogenated caprolactam entering said distilling is less than 50 ppm, preferably less than 10 ppm and more preferably less than 1 ppm. The operation to free the hydrogenated caprolactam mixture from nickel may be any operation known to one skilled in the art for removing nickel from a solution. Preferably, separating nickel from the hydrogenated caprolactam is effected using filtration. An example of a suitable filter is a guard filter. In this embodiment of the invention, said distilling can be performed during a prolonged period of operation without having to remove nickel from the distillation column while ΔPAN_{Ni} remains lower than 3. Depending on the content of nickel in the caprolactam entering said distilling, the distilling can be carried out without interruption for removing nickel from the distillation column during a period of at least 1 month, preferably 1 year and more preferably 2 years.

In a third and even more preferred embodiment of the invention, both the first and second embodiment are applied.

The caprolactam to be purified can be prepared by the Beckmann rearrangement of cyclohexanone oxime in oleum as for example described in DE-A-2508247 or other preparation processes, such as for instance the rearrangement reaction in the presence of an acid ion exchanger as described in GB-A-1342550. Caprolactam obtained by depolymerisation of nylon 6, as for example described in US-A-5169870, can also be purified advantageously using the process of the present invention. It is particularly advantageous to use aqueous solutions of caprolactam obtained starting from cyclohexanone oxime, prepared by oximation of cyclohexanone with hydroxylammonium salts, by a Beckmann rearrangement of the cyclohexanone oxime in the presence of concentrated sulfuric acid, subsequent neutralisation with ammonia resulting in a solution of caprolactam in water and a solution of ammonium sulfate in water, removal of lactam by subjecting the neutralized rearrangement mixture or the solution of caprolactam in water to extraction with an aromatic hydrocarbon like benzene or toluene, optionally washing the obtained organic caprolactam solution with water or an aqueous alkaline solution resulting in a washed solution, and subsequent removal of the organic solvent by for example evaporation or distillation.

The invention will now be elucidated with reference to the following non-limiting examples. Nickel content was determined using Flame Absorption Atomic

Spectrometry.

Comparative Experiment A

At a flow rate of 90.000 kg/hour an aqueous caprolactam mixture, containing 38 wt.% caprolactam, 6 ppm UCL-1 (relative to caprolactam) and 62 wt.% water, is subjected to a hydrogenation.The hydrogenation is carried out in a reactor vessel with a total reaction volume of 15 m^3 with a RaNi catalyst concentration of 15 % at a temperature of 90 °C. The catalyst is suspended in the mixture to be hydrogenated by agitation. Hydrogen feed is controlled such that the hydrogen pressure is 0.5 MPa. The hydrogenation reactor effluent contains less than 2 ppm UCL-1 (relative to caprolactam) and is allowed to have a first separation by sedimentation in a vessel of 15 m^3 volume from which the decanted upper liquid layer is sent to a filtration unit. This filtration unit embodies a set of 2 identical cricket filters equipped with woven cotton cloth with mesh size around 15 μ. Before taking a filtration unit into service a small volume of reactor effluent is being circulated over the filter during 2 hours to build up a cake layer on the filter cloth. Operation of the unit is continued until the pressure drop over the unit exceeds 300 kPa, then a back flush is carried out and the catalyst material is sent back to reactor.

Subsequently, water was removed by distillation to such an extent that the caprolactam contains 0.5 wt.% water. Subsequently, 30.000 kg/hour of this caprolactam is fed to a vacuum distillation column with a diameter of 2.5 m fitted with two beds of Mellapak 250Y packing each having a height of 4m. The applied pressure at the bottom of the column is 3 kPa and the temperature at the bottom of the column is 160 °C.

After a period of three months in operation, an increase of the PAN number of caprolactam over the distillation column has been observed. The increase was 3.5 PAN points. It has surprisingly been found that this increase was for at least a substantial part caused by accumulation of nickel in the distillation column. To remove nickel accumulated in the column, the run was stopped and the column was flushed with 5.4 m^3 of a 10% nitric acid solution by circulating the nitric acid solution over the internals and reboiler during a period of 4 hours. The nickel content in the nitric acid liquid resulting from this operation was analyzed using Flame Atomic Absorption Spectrometry. From this analysis, it could be derived that 300 kg of nickel had been accumulated in the distillation column. After having removed nickel from the distillation column, the run was continued. The PAN number of caprolactam over the distillation column did not show any increase.

The performance of the filtration units was evaluated by analyzing weekly the Ni content of the caprolactam stream leaving the filtration unit. Over a period of three month the average Ni content was over 2 ppm. In the caprolactam entering the distillation column the average Ni content was 6 ppm. The Ni content was analyzed using Flame Atomic Absorption Spectrometry.

Example 1

Comparative Experiment was repeated, except that the dehydrogenated, dewatered caprolactam was filtered before entering the distillation column. An additional filter unit was installed in front of the distillation column. The unit contains two parallel cartridges with 10 μ mesh being alternately in service to ensure continuous operation.. The nickel content in the caprolactam stream leaving this filtration unit was 1 ppm in average (measured with Flame Atomic Absorption Spectrometry).
After a period of half a year in operation, the PAN numbers gradually climbed from 2.5 to 3.5. It was decided to clean the distillation column again in an identical manner as under comparative experiment A. It turned out that 80 kg nickel had been fouling the column.

Example 2

Example 1 was repeated except that the cartridges in the filtration unit in front of the distillation column were replaced by a type of finer mesh (5 μ). Nickel content in the filtrate was tracked over a period of time resulting in figures well below 1 ppm (100 ppb). After 2 years service, the PAN numbers measured in the caprolactam streams leaving this column show an increase of 0.3. The column has been cleaned after this period in service in the same way as under Comparative Experiment A. It has been found that the nickel accumulation in the column was no more than 4 kg.

Example 3

Comparative experiment was repeated, except that the slurry-phase hydrogenation was replaced by a fixed bed hydrogenation. The fixed-bed type catalyst was a nickel on alumina catalyst. The catalyst load to the hydrogenation reactor was 9 ton of an alumina based Ni catalyst. The nickel content is 25%, the particle size 3.2

mm. The hydrogen pressure applied is 0.5 MPa; the hydrogenation was carried out at a temperature of 90 °C. The hydrogenated caprolactam leaving the fixed bed hydrogenation was subjected to the dewatering and subsequently the hydrogenated, dewatered caprolactam was fed to the distillation column. After 1 year in operation an increase in PAN number over the distillation column of 1 point (from 2.5 to 3.5) gave rise to clean the distillation column again in the way as under Comparative Experiment A. It has surprisingly been found that still 52 kg Ni had been fouling the column.

Example 4

Example 3 was repeated except that a filtration unit was installed in front of the distillation column consisting of two parallel cartridges, of which one is in use, having a mesh of 5 μ. After more than two years in operation no significant increase in PAN numbers over the column could be detected. Incidental analysis of nickel content of the hydrogenation reactor effluent stream showed very low figures (around 10-30 ppb).

CLAIMS

1. Process for purifying caprolactam, said process comprising
   (a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst,
   (b) distilling at least a portion of the hydrogenated caprolactam in a distillation column, characterized in that the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 3$, wherein
   $\Delta PAN_{Ni} = \Delta PAN - \Delta PAN_{Ni=0}$,
   ΔPAN = increase of the PAN number of caprolactam during distilling,
   $\Delta PAN_{Ni=0}$ = increase of the PAN number of caprolactam during distilling under the same conditions in a distillation column free of nickel.
2. Process according to claim 1, wherein the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN \leq 3$.
3. Process according to claim 1, wherein the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 2$.
4. Process according to claim 3, wherein the distillation column contains nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 1$.
5. Process according to any one of claims 1-3, wherein said distilling is performed continuously.
6. Process according to any one of claims 1-5, wherein the process further comprises, prior to said distilling, separating nickel from the hydrogenated caprolactam.
7. Process according to claim 6, wherein said separating is effected using filtration.
8. Process according to any one of claims 1-7, wherein the nickel containing hydrogenation catalyst is a fixed bed catalyst.
9. Process according to any one of claims 1-8, wherein the nickel containing hydrogenation catalyst particles are suspended in the caprolactam to be hydrogenated.
10. Process according to claim 9, wherein after said hydrogenation the catalyst particles are separated from the hydrogenated caprolactam.
11. Process according to claim 6 and 10, wherein said separating of nickel from

hydrogenated caprolactam is effected after said separating of catalyst particles from the hydrogenated caprolactam.

12. Process according to any one of claims 1-11, wherein said distilling is effected in a distillation column having a bottom temperature of between 110 and 180 °C.

13. Process according to any one of claims 1-12, wherein said distilling is effected in a distillation column in which the caprolactam of the hydrogenated caprolactam has a residence time higher than 5 minutes.

14. Process according to any one of claims 1-13, wherein water is separated from the hydrogenated caprolactam prior to said distilling.

15. Process according to claim 6 or 7 and 14, wherein separating nickel from the hydrogenated caprolactam is effected after said separating of water and prior to said distilling.

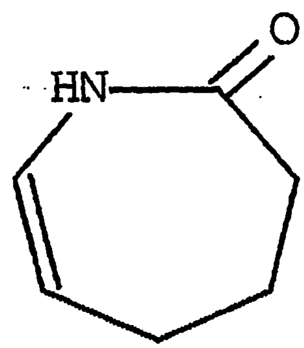
ABSTRACT

The invention relates to a process for purifying caprolactam, said process comprising

(a) subjecting the caprolactam to a hydrogenation by treating the caprolactam with hydrogen in the presence of a heterogeneous nickel containing hydrogenation catalyst,

(b) distilling at least a portion of the hydrogenated caprolactam in a distillation column containing nickel in an amount sufficiently low such that $\Delta PAN_{Ni} \leq 3$, wherein $\Delta PAN_{Ni} = \Delta PAN - \Delta PAN_{Ni=0}$,
ΔPAN = increase of the PAN number of caprolactam during distilling, $\Delta PAN_{Ni=0}$ = increase of the PAN number of caprolactam during distilling under the same conditions in a distillation column free of nickel.

Formula sheet



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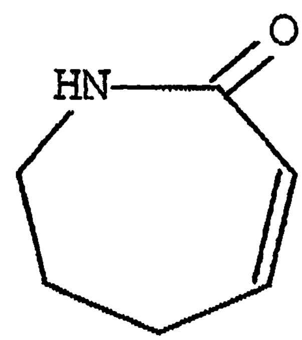

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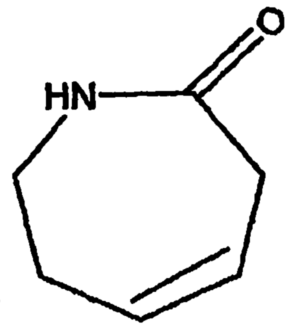

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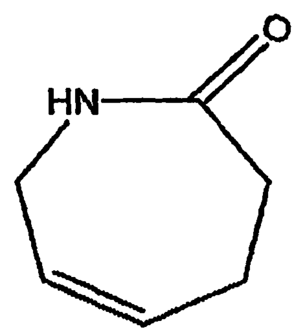

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